Determination of pH of an aqueous solution of mainstream cigarette smoke using a routine linear analytical smoking machine

1 Scope

This procedure specifies the method for trapping mainstream cigarette smoke in degassed water and subsequent pH analysis of the water solution and glass fiber filter pad. The mainstream cigarette smoke is generated and collected using a linear analytical smoking machine.

2 Normative references

At the time this procedure was written, the editions indicated were valid.

ISO 3308:1991. Routine analytical cigarette - smoking machine - Definitions and standard conditions.

ISO 3402:1991. Tobacco and tobacco products - Atmosphere for conditioning and testing.

ISO 7210: 1997. Routine analytical cigarette - smoking machine - Additional test methods.

ISO 8243:1991. Cigarettes - Sampling.

Federal Register, Volume 32, No.147, p. 11178, August 1, 1967.

Federal Register, Volume 45, No. 134, p. 46483, July 10, 1980.

105 CMR 660.000 Cigarette and Smokeless Tobacco Products: Reports of Added Constituents and Nicotine Ratings, Massachusetts Department of Public Health, August 19, 1997.

3 Definitions

For the purpose of this procedure, the following definitions apply.

3.1 pH: A measure of the acidity or basicity of an aqueous solution measured as the negative logarithm of the activity of hydrogen ions.

3.2 total particulate matter: That portion of the mainstream smoke which is trapped in the smoke trap, expressed as milligrams per cigarette (mg/cigt.).

3.3 clearing puff: Any puff taken after the cigarette has been extinguished and removed from the cigarette holder.

3.4 smoking process: The use of a smoking machine to smoke cigarettes from lighting to final puff.

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3.5 laboratory sample: The sample intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample.

3.6 conditioning sample: The cigarettes selected from the test sample for conditioning prior to tests for total particulate matter yield.

3.7 test portion: A group of cigarettes prepared for a single determination and which is a random sample from the test sample or conditioned sample as appropriate.

3.8 conditioned sample: Conditioned cigarettes for total particulate matter yield tests.

4 Principle

Sampling of the test cigarettes. Conditioning of the test cigarettes. Smoking of test cigarettes on a linear analytical smoking machine equipped with a degassed water containing trap with simultaneous collection of mainstream smoke in the water and total particulate matter in a glass fiber filter trap. Combination of total particulate matter on the glass fiber filter and aqueous solution so collected. Determination of pH of the aqueous solution containing the glass fiber pad.

5 Apparatus and reagents

Normal laboratory apparatus and reagents and in particular the following items:

5.1 Smoke trap apparatus. Manufactured by Research Glass, Richmond, VA, part number pm030496. The apparatus consists of two glass traps, with 24/40 ground glass fitting, with a volume of approximately 90 mL each, attached in series using glass ball joints. The cigarette is inserted into a rubber cigarette holder that is mounted on a 0.5 inch diameter, medium wall glass tube, (referred to as the impinger inlet tube). This glass tube has a 90° bend at a distance of 125 mm from the rubber cigarette holder, (this allows placement of the apparatus within the smoking machine hood behind the Filtrona 350 smoking bar / ashtray assembly with the glass tube passing through the smoking bar). Glass ball joints are used to attach the glass tube to the first impinger. An approximately two inch section of ¼ inch I.D. polyvinyl chloride (PVC) tubing is attached to the exit end of the second impinger. The other end of the PVC tubing is inserted, to a 9 mm insertion length, into the harmonized filter holder that is mounted onto the carrier eccentrics, carrier slide, and PVC tubing assembly that is reassembled behind the smoking bar.

NOTE 1 A drawing of the smoking trap apparatus is given in Figure 1.

NOTE 2 All glassware is washed prior to each use with a laboratory soap solution, (Alconox or equivalent), and dried at 100°C for a minimum of 3 hours.

5.2 Filtrona 350 smoking machine. Modified to comply with the requirements of ISO 3308. The filter carrier slide is removed from the smoking bar for the port(s) utilized for the smoking portion of this procedure.

NOTE 2 Due to the positioning of the smoke trap apparatus, (5.1), within the smoking machine hood, a maximum of two ports of the twenty available smoking machine ports were simultaneously utilized within

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this procedure. No criteria exist for which two ports are utilized except for smoke trap spacing. Ports 5 and 14 were utilized within this procedure.

Figure 1 - Picture of the pH smoke trap apparatus. Where: A is the rubber cigarette holder; B is the impinger inlet tube; C is the glass ball joint with clamp; D is the harmonized glass filter pad holder with PVC tubing inserted; E is the Filtrona carrier eccentric; and, F is the Filtrona carrier slide.

5.3 44 mm glass fiber filter pads. Manufactured by Whatman International Ltd. and certified to comply with the requirements of ISO 3308.

5.4 Filtrona harmonized filter pad holders. Manufactured by Filtrona to comply with the requirements of ISO 3308.

5.5 Omnidirectional air velocity probe and meter. Manufactured by Schiltknecht.

5.6 Soap bubble flow meter. Manufactured by Borgwaldt, graduated at 40 to 80 mL with a resolution of 0.2 mL.

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5.7 **Ruler.** Certified for measurements to the nearest 0.5 mm.

5.8 **pH meter.** Model 25 Accumet pH/ion meter manufactured by Fisher Scientific.

5.9 **Glass body combination electrode with Ag/AgCl internal reference element.** Manufactured by Fisher Scientific.

5.10 **50 mL Optifix solvent dispenser.** Manufactured by EM Science. Calibration of the dispenser is performed daily using a 50 ± 0.2 mL certified graduated cylinder.

5.11 **Degassed water.** HPLC grade water from Fisher Scientific. Prior to use, gas chromatography grade helium gas is bubbled, at a slow continuous stream, through the water for a minimum of 12 hours.

6 Sampling and sample preparation

6.1 Sampling

Remove one cigarette from each pack of the laboratory sample to form the conditioning sample.

6.2 Cigarette marking

The cigarette butt length is determined following FTC protocol as the overwrap, (tipping paper), length plus 3 millimeters. Per ISO 3308, the overwrap lengths of twenty cigarettes are measured to an accuracy of 0.5 mm and the average is determined to an accuracy of 0.5 mm. The 9 mm insertion and determined butt length are marked on each cigarette using a soft tip pen to avoid damage to the cigarette.

NOTE 3 Butt length is defined in ISO 3308 as the length of unburnt cigarette remaining at the moment when smoking is stopped.

NOTE 4 If the determination of butt length has been performed on the laboratory sample as part of the total particulate matter determination, (ISO 4387), this determination does not need to be repeated on this test sample.

6.3 Cigarette taping

One-half of the cigarette overwrap is covered with Number 600, Scotch Brand tape. The width of the tape used is one-half the known circumference of the cigarette brand. The tape is applied lengthwise over the cigarette overwrap starting at the cigarette rod end and ending flush with the mouth end of the cigarette. Any tape extending past the end of the cigarette is cut off using a razor blade.

NOTE 5 For cigarettes of 24.8 to 25.2 mm circumference, the theoretical width of applied Scotch tape should be 12.4 to 12.6 mm. Commercially available, one-half inch (12.7 mm) Scotch tape is used due to the inability to cut or purchase the tape exactly to the width required (± 0.1 mm). The actual coverage of the overwrap for cigarettes of these circumferences is thus slightly greater than one-half. The intent of this

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process is to cover one-half of the cigarette ventilation holes and this variance in tape width reflects ventilation hole coverage slightly more than one-half.

6.4 Sample equilibration

The cigarettes are placed in an equilibration tray. This conditioning sample is equilibrated in a conditioning environment specified in ISO 3402 for a minimum of 48 hours and a maximum of 10 days.

7 Standard smoking conditions

The standard smoking conditions used are in accordance with ISO 3308:1991. 4 except for the following deviations specified per 105 CMR 660.000.

7.1 Puff volume

The standard puff volume measured in series with a pressure drop of 1 kPa is 45 ± 0.4 mL, (see Note 6). In order to achieve a puff volume of 45 mL at the entrance end of the smoke trap apparatus, the smoking machine is set at a puff volume of 47.5 mL as measured from the harmonized filter pad holders as described in ISO 4387:1991. 7.5.3.3.

NOTE 6 ISO 4387:1991. 7.5.3.3 states that puff volume be set to 35 mL ± 0.3 mL. Due to the larger puff volume utilized in this procedure and graduation of the appropriate soap bubble meter, (0.2 mL), a puff volume range of ± 0.4 mL was applied. This acceptable volume range, 0.8 mL for 45 mL is consistent with 0.6 mL for 35 mL based on percent acceptable variation per volume measured.

7.2 Puff frequency

The standard puff frequency is one puff every 30 seconds with a standard deviation of not greater than 0.5 seconds.

8 Smoking process

8.1 Laboratory environment

The testing atmosphere in the laboratory where the smoking is carried out is in accordance with ISO 3402.

8.2 Smoking plan

The test portion is comprised of two cigarettes randomly selected from the conditioning sample which comprises one determination. Three replicate test portions are utilized to generate the reported pH result.

8.3 Smoking process

The smoking machine set up is in accordance with ISO 4387 except for those changes noted in clauses 5.1 and 5.2 and section 7. Fifty mL of degassed water is dispensed, (see clause 5.10), into each impinger trap. Each impinger is secured with a 24/40 ground glass fitting clamp. The

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smoke trap apparatus is assembled per clause 5.1. All glass ball joints are secured with clamps. String is applied to the puff termination device on the smoking machine per ISO 4387 for those ports being utilized. A test cigarette is inserted to the insertion mark into the rubber cigarette holder. The smoke trap apparatus is positioned to insure that the puff termination string crosses the butt length mark on the test cigarette. The balance of the smoking run is conducted in accordance with ISO 4387:1991. 7.5.4.

After completion of the test sample, two cigarettes per determination, two clearing puffs are taken. After the last clearing puff, a timer is activated to track the time for the pH determination. The aqueous solution from each impinger tube is combined in a 150 mL beaker containing a magnetic stirring bar. The glass fiber pad is removed from the filter pad holder and added to the beaker. A disposable dropper is used to rinse the impinger inlet tube (see clause 5.1) with three aliquots of the resultant aqueous solution. The activated timer and the beaker containing the aqueous solution so collected and glass fiber pad so collected are utilized for the pH determination.

NOTE 7 The above process is utilized to generate a process blank. The same process is applied except 11 puffs are drawn through two unlit cigarettes and two clearing puff after the second cigarette. Industry Monitor cigarettes are utilized.

NOTE 8 The above process is performed as documented above using Industry Monitor cigarettes. The monitor cigarette is tested on each smoking port at the beginning of the testing session. The monitor results are used to ensure consistency of the smoking and pH processes.

9 pH determination

9.1 Daily pH calibration

Calibration of the pH meter is performed at the beginning of each daily testing session. Calibration is performed as a two point standardization using pH 4 and pH 10 buffers. The slope or efficiency must be within 95 to 105% before proceeding.

9.2 Calibration standard check

A calibration standard check is performed using a pH 7 buffer before each test sample measurement. The buffer response must be 7.00 ± 0.10 pH units or recalibration is required.

9.3 pH determination of test samples

The beaker containing the so collected aqueous solution, glass fiber filter pad, and magnetic stirring bar from clause 8.3 is placed on a magnetic stirrer. At or before 10 minutes after the last clearing puff, as indicated by the activated timer, the pH electrode is submerged into the test solution. At 15 ± 0.5 minutes after the last clearing puff, the pH value is recorded.

NOTE 9 The pH meter is interfaced to a personal computer which allows automatic transmission of data from the pH meter into an Excel spreadsheet utilizing an Excel macro.

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10 Test report

The average of three replicate determinations from the conditioning sample is reported to 0.01 pH units.

The appropriate information concerning the characteristic data about the cigarette, sampling, and description of test is recorded in accord with ISO 4387.

11 Repeatability and reproducibility

The industry monitor cigarette was analyzed twice per day for seven days. The mean pH value for Industry Monitor #16 was 5.35 pH units. All obtained individual data values, for cigarette samples analyzed per the method, ranged from 5.2 to 5.5 pH units thus placing the monitor results at the midpoint of the sample range.

From the industry monitor results, the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value (r = 0.08 pH units) on average not more than once in 20 cases in the normal and correct operation of the method.

Due to time limitations on implementation of the method, a collaborative study involving a suitable number of laboratories could not be performed for the determination of the method reproducibility (R) value.

12 Revision History

12.1 December 1997

Original procedure written by C. H. Callicutt and J. M. Garman.

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